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One Pot Synthesis of 4H-Pyran Derivatives by Use of Efficient and Recyclable Catalyst

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ABSTRACT

Background: Pyrans are one of the most significant skeletons of oxygen-containing heterocyclic molecules, which exhibit a broad spectrum of medicinal applications. Green one-pot methodologies for synthesizing these heterocyclic molecules have received significant attention. The paper focusses on a simple approach to synthesize 4H-pyran derivatives by one pot multi-component reaction of aromatic aldehyde, β-ketoester or β-diketone with the use of Nd_2O_3 as recyclable and efficient catalyst.

Methods: The reagents ethyl acetoacetate (2mmol) and 4-chlorobenzaldehyde (1mmol) and varying amount of L-proline or Nd_2O_3 catalyst were stirred or refluxed in PEG-400, water or H_2O/EtOH (1:1) solvent to yield 4H pyran. The reaction time and yield of the product was monitored. 20 mmol of Nd_2O_3 catalyst was sufficient to catalyze the reaction in good yield. The optimized conditions of the reaction were effectively implemented to synthesise various 4H pyran derivatives. The synthesized compounds were characterized and confirmed by different spectroscopic techniques like ^{1}H NMR and FTIR. The values reported are well in agreement with the expected values.

Results: The results yielded lesser reaction time of around 30 to 54 mins with a yield of around 82 to 94 %. The reaction required only 20mmol catalyst in (1:1, v/v, ml) of H_2O/C_2H_5OH mixed solvents. It demonstrates the low cost, simple work up process using H_2O/ C_2H_5OH as greener solvents.

Conclusions: Neodymium (III) oxide (Nd_2O_3) is a powdered rare earth metal oxide used as a novel catalyst which can be recovered through simple filtration, rinsed with hot ethanol, and dried at 150^0C for further use. The recovered catalyst can be reused successfully at least three times with minimal loss in activity.

Keywords: Recyclable Catalyst; Green Solvent; 4H-Pyran Derivative; Multicomponent Reaction; Nd_2O_3; Synthesis; Ecofriendly; Heterocyclic Molecules; One Pot Synthesis; Cost Effective.

1. Introduction

Organic chemists continuously investigate innovative synthetic approaches utilizing novel reagents and catalysts to facilitate chemical transformation. The development of environmentally benign chemical processes and technologies is the basic need of green chemistry. Eco-friendliness of the chemical process is increased, if the multi-component approach is utilized. The quick assembly of molecular diversity through multicomponent reactions (MCRs) has earned significant attention, particularly in the creation of heterocyclic, drug-like libraries.

In particular, one-pot synthetic methodology protocols under green conditions of heterocyclic analogues have become a sizable alternative in modern organic chemistry to conventional multistep synthesis. Because of the improved productivity and substantial time saving, multicomponent reactions (MCRs) are recognized as reliable synthetic green methodologies [1,2]. Several advantages of MCRs driven by green credentials are crucial in organic synthesis and drug discovery programs [3,4]. The multicomponent reactions (MCRs) have been considered in synthesis of organic, combinatorial, bioactive medicinal and heterocyclic compounds [5-7], because of the compact reactions, easy and simply procedures and good yields. Advantages of MCRs are wide including simple and uncomplicated protocols, inexpensive reactants and green considerations [8-9]. The additional benefits of MCRs are shorter reaction times and application of green solvent and in general has cost-effective and environmental friendly approach [10].

Compounds with pyran ring system possess many pharmacological properties and plays important role in biochemical processes [11]. A few recent work on Multicomponent synthesis of 4H-pyran derivatives was performed using KOH loaded calcium oxide as catalyst in solvent free condition [12]. The method uses dicyanomethane, a toxic substance in the synthesis of 4H pyran. Pyran derivatives synthesized using green, catalyst free method supported by ultrasonic irradiation and microwave assistance are reported [13,14]. The method reported using ultrasonic irradiation suffer challenges in achieving reproducible results. The microwave assisted method uses ethanol as solvent in synthesis which can be explosive at the experimental conditions. MCRs were also carried in the presence of magnetic LDH as a nanocatalyst [15]. Magnetic LDH nanocatalyst suffered drawback of multiple step synthesis in preparation of catalyst and costly characterization of synthesized catalyst. Few pyran derivatives were synthesized using reusable ionic liquid and recyclable basic phase-transfer catalyst [16,17]. The synthesis suffered drawback because of the use of methylene cyanide, a toxic reagent.

A novel recyclable hydrolyzed nanomagnetic copolymer catalyst was used for green, and one pot synthesis of tetrahydrobenzo[b]pyans [18]. The nanomagnetic copolymer catalyst Fe_3O_4- Hydrol-PMMAn, uses benzoyl peroxide in synthesis, which breaks down into benzene, a known carcinogen, at high temperatures. Also the characterization uses EDS, TEM, SEM & XRD techniques which are expensive. A review on recent progress in the Multicomponent synthesis of pyran derivatives by sustainable catalysts under green conditions by Maddila et al [19] demonstrates use of toxic malonitrile as a reagent for synthesis and nanomaterial catalysts which have time consuming workup procedures and costly characterization.

It is observed that numerous existing methods face drawbacks of utilizing toxic reagents, use of explosive solvents, expensive synthesis and characterization of the catalyst and tedious work-up procedure. Moreover, in the most of reported method catalysts are not recyclable.

Hence there is a need to develop an efficient and recyclable catalyst for the synthesis of 4H pyran derivatives. Neodymium (III) oxide (Nd_2O_3) is one such catalyst which is a powdered rare earth metal oxides of light blue color having a significantly high melting point 2233^{o}C and is inexpensive and is easily available.

Therefore, in this correspondence, we aim to disclose Nd_2O_3 as a reusable catalyst in the one-pot synthesis of 4H-pyran derivatives in the aqueous medium.

1.1. Objectives of the Study

The following are the objectives of this study:

(1) To promote sustainability of the catalyst used.

(2) To reduce waste, enhance efficiency and lower costs by enabling repeated use of catalysts.

(3) To minimize environmental impact and resource consumption.

(4) To perform multiple steps of synthesis in a single reaction vessel, avoiding lengthy purification and separation process.

(5) To use greener solvents in order to resulting in minimizing and eliminating hazardous chemicals.

2. Methods

Chemicals were sourced from SBL, Loba Chemie, and Ottokemi. The structural verification of the derivatives was conducted utilizing FTIR & NMR spectroscopy. FTIR was done with KBr pellet technique on a Bruker 3000 Hyperion Microscope fitted with a Vertex 80 FTIR system (Germany). ^{1}H NMR spectra was acquired in deuterated dimethyl sulfoxide (DMSO-d_6) at 600 MHz with a JEOL ECZR Series 600 mega Hertz NMR Spectrometer (Japan), with TMS as internal standard, and chemical shifts were recorded in δ ppm.

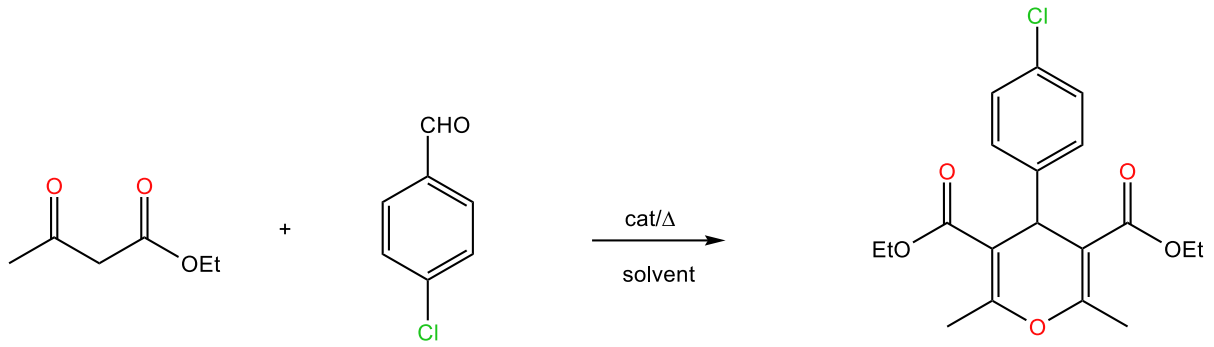
2.1. Common preparation technique for synthesis of 4H-pyran derivatives

Aromatic aldehyde (1mmol), β-ketoester or β-diketone (2 millimol) and catalyst Nd_2O_3 (10 millimol) was refluxed in water (2 ml) until the reaction reached completion (TLC). The resulting mixture was subjected to extraction with dichloromethane (10 ml) to recover the Nd_2O_3 catalyst from aqueous layer. Solvent was removed on rotary evaporator and products were purified utilizing column chromatography with a combination of n-hexane and ethyl acetate (7:3).

3. Results

3.1. Optimization of reaction conditions

We made an initial attempt to synthesize Diethyl-4-(4-Chlorophenyl)-2,6-dimethyl-4*H*-pyran-3,5-dicarboxylate in various solvent using L-Proline or Nd_2O_3 catalyst in varying concentration (Scheme 1). During optimization of reaction condition (Table 1), a mixture of ethylacetoacetate (2 equiv.), 4-chlorobenzaldehdye (1 equiv.), polyethylene glycol (PEG-400, 2 ml) and catalyst L-proline (10 equiv.) was kept for stirring at room temperature for 180 min and did not afford even trace amount of desired product. After stirring the reaction mixture at 80^{0}C for 180 min using L-proline as catalyst (10, 20, 40 equiv.), offered the yield of the desired product (30%, 32%, 35%) respectively (entries 2-4). Next, we started to use water as the solvent under varying temperatures using a catalyst Nd_2O_3, offered appreciable amount of expected product (entries 5-7). However, a better yield was obtained with mixed solvents H_2O/C_2H_5OH (1:1, v/v, ml) (entries 8 and 9). Encouraged by these observations, we proceeded to optimize the amount of catalyst and found that 20 mmol of Nd_2O_3 is sufficient to catalyze the reaction (entries 10-12).



Ethylacetoacetate 4-chlorobenzaldehdye Diethyl-4-(4-Chlorophenyl)-2,6-dimethyl-4*H*-pyran-3,5-dicarboxylate

Scheme 1. Synthesis of Diethyl-4-(4-Chlorophenyl)-2,6-dimethyl-4*H*-pyran-3,5-dicarboxylate

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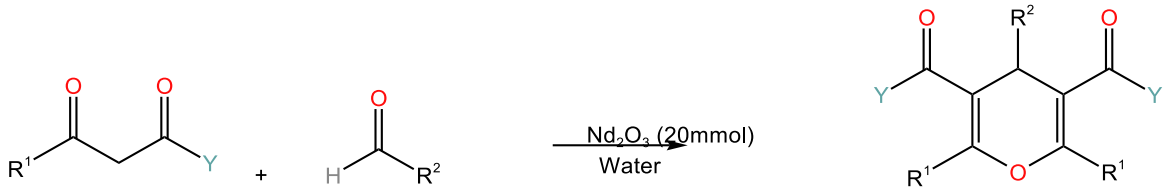
Table 1. Optimization of reaction condition[a]

Entry	Catalyst (mmol)	Solvent	Temp (^oC)	Time (min)	Yield
1	L-Proline (5)	PEG-400	Room Temp	180	-
2	L-Proline (10)	PEG-400	80	180	30
3	L-Proline (15)	PEG-400	80	180	32
4	L-Proline (20)	PEG-400	80	180	35
5	Nd_2O_3 (5)	H_2O	80	180	40
6	Nd_2O_3 (5)	H_2O	reflux	180	50
7	Nd_2O_3 (10)	H_2O	reflux	180	60
8	Nd_2O_3 (5)	H_2O/EtOH (1:1)	80	180	70
9	Nd_2O_3 (5)	H_2O/EtOH (1:1)	reflux	180	78
10	Nd_2O_3 (10)	H_2O/EtOH (1:1)	reflux	45	92
11	Nd_2O_3 (15)	H_2O/EtOH (1:1)	reflux	45	92
12	Nd_2O_3 (20)	H_2O/EtOH (1:1)	reflux	45	93

[a]ethyl acetoacetate (2mmol), 4-chlorobenzaldehyde (1mmol), solvent (2ml) [b]Isolated yield.

3.2. Synthesis of 4H-Pyrans under optimum reaction condition

Using the best condition reported in Table 1, we then continued to study the condensation reaction of β-ketoester or β-diketone with a variety of aromatic aldehydes by reflux method (Scheme 2).



1 2 3(a-j)

Scheme 2. General reaction for synthesis of 4H-Pyrans

Diethyl 4-(4-chlorophenyl)-2,6-dimethyl-4H-pyran-3,5-dicarboxylate **(3a):** mp 96^{o}C: FTIR (cm^{-1}): 3050, 1725, 1610, 1305, 785; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.00 (d,2H), 4.19 (q, 4H), 3.94 (s,1H), 1.30 (t,6H).

Diethyl 2,6-dimethyl 4-(4-nitrophenyl) 4H-pyran-3,5-dicarboxylate **(3b)**: mp 193^{o}C; FTIR (cm^{-1}): 3054, 1720, 1607, 1520, 1315; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.32 (d, 2H), 4.19 (q,4H), 3.94 (s,1H), 1.30(t,6H).

Diethyl 4-(4-methoxyphenyl)-2,6-dimethyl-4H-pyran-3,5-dicarboxylate **(3c)**: mp 112^{o}C; FTIR (cm^{-1}): 3047, 1723, 1617, 1523, 1305; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.00 (d,2H), 4.19 (q, 4H), 3.94 (s,1H), 3.52(3H), 1.30 (t,6H).

Diethyl 2,6-dimethyl 4-(3-nitrophenyl) 4H-pyran-3,5-dicaroxylate **(3d)**: mp 194^{o}C; FTIR (cm^{-1}): 3050, 1752, 1613, 1518, 1302; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.40(t, 2H), 7.99(s, 1H), 7.45(d, 1H), 4.19(q, 4H), 1.71(s, 6H), 1.30 (t, 6H).

Diethyl 2,6-dimethyl- 4-phenyl- 4H-pyran-3,5-dicaroxylate **(3e)**: mp 96^{o}C; FTIR (cm^{-1}): 3040, 1755, 1603, 1510, 1307; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.06(d, 2H), 4.19(q, 4H), 3.94 (s, 1H), 1.30 (t, 6H).

Dimethyl 4-(4-chlorophenyl)-2,6-dimethyl- 4H-pyran-3,5-dicaroxylate **(3f)**: mp 264^{o}C; FTIR (cm^{-1}): 3053, 1748, 1608, 1510, 1313, 540; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.00 (d,2H), 4.19 (q, 4H), 3.94 (s,1H), 1.30 (t,6H).

Dimethyl 4-(4-methoxyphenyl)-2,6-dimethyl- 4H-pyran-3,5-dicaroxylate **(3g)**: mp 94^{o}C; FTIR (cm^{-1}): 3059, 1739, 1616, 1504, 1310; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.00 (d,2H), 4.19 (q, 4H), 3.94 (s,1H), 3.52(3H), 1.30 (t,6H).

Diethyl 2,6-dimethyl 4-phenyl 4H-pyran-3,5-dicaroxylate **(3h)**: mp 94^{o}C; FTIR (cm^{-1}): 3040, 1746, 1624, 1503; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.06(d,2H), 7.07 (t,1H), 3.76 (s, 3H), 3.94 (s,1H), 1.71 (t,6H).

Dimethyl 2,6-dimethyl (4-methoxyphenyl) 4H-pyran-3,5-dicaroxylate **(3i)**: mp 112^{o}C; FTIR (cm^{-1}): 3038, 1750, 1624, 1507, 1310; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.06(d,2H), 7.07 (t,1H), 3.76 (s, 3H), 3.94 (s,1H), 3.56(3H), 1.71 (t,6H).

Dimethyl 2,6-dimethyl (4-chloro phenyl) 4H-pyran-3,5-dicaroxylate **(3j)**: mp 94^{o}C; FTIR (cm^{-1}): 3054, 1748, 1604, 1510, 785; ^{1}H NMR (600 mHz, DMSO-d_6, δ-ppm): 7.15(m), 7.00 (d,2H), 3.94 (s,1H), 1.71 (t,6H).

Table 2 demonstrates the synthesis of 4H-Pyran derivatives under optimized reaction condition.

Table 2. Synthesis of 4H-Pyrans under optimum reaction condition

Compounds	Y	R^1	R^2	Time (mins)	Yield[a] (%)	M.P[b] (^{o}C)
3a	OEt	Me	4-ClC_6H_4	45	92	96
3b	OEt	Me	4-$O_2NC_6H_4$	30	94	193
3c	OEt	Me	4-$MeOC_6H_4$	52	82	112
3d	OEt	Me	3-$O_2NC_6H_4$	34	89	194
3e	OEt	Me	C_6H_5	35	84	96
3f	OMe	Me	4-ClC_6H_4	48	91	264
3g	OMe	Me	4-$MeOC_6H_4$	54	82	94
3h	OMe	Me	C_6H_5	35	83	94
3i	Me	Me	4-$MeOC_6H_4$	34	85	112
3j	Me	Me	4-ClC_6H_4	45	88	94

[a]Isolated yield [b]Melting points were taken in open capillary bath and are uncorrected.

As seen in Table 2, the optimized conditions for the reaction were effectively implemented to various substrates. Whether the aromatic aldehyde contain electron with drawing or electron donating substituents, Nd_2O_3 efficiently promoted the reaction with good to excellent yields.

3.3. Comparative analysis

Table 3 demonstrates the effects of different catalyst on the synthesis of 4H-pyran derivatives as cited in Rajput et al [20].

Table 3. The effects of different catalyst on the synthesis of 4H-pyran derivatives

Entry	Catalyst (mol %)	Time	Yield %
1	Acetic acid	12 hour	Intermediate
2	Piperidine	2 hour	40
3	Catalyst free	12 hour	No reaction
4	Al_2O_3	8 hour	50
5	MgO	10 hour	60
6	CaO	7 hour	42
7	Nd_2O_3	45 min	93

The synthesis of 4H pyran in presence of acetic acid as catalyst required 12 hours for the reaction to complete with an intermediate yield (entry 1). The same reaction was carried out in presence of pipredine as catalyst and the product was formed in 40% yield after 2 hours (entry 2). When reaction was carried out without catalyst no product was found after 12 hours (entry 3). However when the reaction was carried out in the presence of various catalysts like Al_2O_3, MgO and CaO, the product was formed in 50%, 60%, and 42% yield respectively requiring 8 hours, 10 hours and 7 hours respectively (entry 4, 5, 6).

However, when the reaction was carried in presence of Nd_2O_3, the reaction required 45 mins for completion, with a yield of 93% (entry 7). Hence it confirms Nd_2O_3 to be highly efficient catalyst in the synthesis of 4H pyran.

3.4. Reutilization of recovered catalyst (Nd_2O_3)

The catalyst Nd_2O_3 was recollected through simple filtration, rinsed with hot ethanol, and dried at 150^0C for the further use. To investigate activity of the recovered catalyst, it was utilized again in the model reaction. The results shown in Table 4 indicates that the recovered catalyst (Nd_2O_3) can be reused a minimum of three times with minimal loss of activity.

Table 4. Reutilization of recovered catalyst (Nd_2O_3)

No of uses	Yield (%)	Recover of Nd_2O_3 (%)
1	97	95
2	93	92
3	90	90

4. Conclusion

In conclusion, we have been successful in developing a simple approach to the synthesis of 4H-pyrans using a novel Nd_2O_3 as recyclable catalyst which can be reused at least three times with minimal loss in activity resulting in lesser cost. Utilizing green solvent like water and ethanol in (1:1 v/v) with a total volume of 2 ml, along with lesser reaction time of around 45 mins, with good product yield of around 93%, and a simple work-up process of refluxing are the merits of the present protocol. The exploration of Nd_2O_3 as catalyst for other multicomponent reactions leading to biologically active compounds is underway. In general, one-pot synthetic methods of oxygen

containing heterocyclic molecules under green conditions have immense scope in modern organic synthesis in future.

5. Future Suggestions

1. Explore the synthesis of diversely substituted 4H-pyran derivatives by using a wider range of aldehydes, active methylene compounds, and other reactants. This could include incorporating functional groups with potential biological activity.

2. Explore multicomponent reactions involving the synthesis of 4H-pyran derivatives with other heterocyclic systems or the incorporation of other functional groups. This could lead to the creation of complex and biologically active molecules.

3. Utilize computational tools to understand the reaction mechanism and identify potential catalysts or reaction conditions that could improve efficiency and selectivity. This knowledge can guide the design of new and more effective catalysts.

4. Test the synthesized 4H-pyran derivatives for potential biological activity, such as antimicrobial, anti-cancer, or antioxidant properties. This research could lead to the development of new and useful pharmaceutical or agricultural agents.

Declarations

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Competing Interests Statement

The authors have not declared any conflict of interest.

Consent for publication

The authors declare that they consented to the publication of this study.

Authors' contributions

All the authors took part in literature review, analysis, and manuscript writing equally.

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